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(54) EXTRACTION PROCESS

(57) An extraction process for extracting a substance from a source containing the substance, for instance fruit, nuts, plants, herbs and drugs, comprises subjecting the source in the presence of an extraction liquid for the said substance, generally chosen from alcohols and water or mixtures thereof, to the influence of ultrasonic radiation, for instance with an acoustic power of 10—100 W, a frequency of 20—50 kHz and a period of 10—100 minutes, the extraction liquid serving as a transmission medium for the ultrasonic energy. Thus vanillin is obtained from line pieces and a vermouth extract is obtained from Torino herb mixture.

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SPECIFICATION

EXTRACTION PROCESS

The invention relates to an extraction process, especially for obtaining extracts useful in the spirituous liquor industry.

In the spirituous liquor industry liqueurs of similar alcoholic beverages are produced wuth the use of drug extracts, these being normally made up of mixtures of different aromatic substances, some of which only form by chemical reactions in the course of the extraction process. These vegetable or drug extracts are generally produced by macerating, digesting or percolating the raw materials by means of ethanol or a mixture of ethanol and water.

From the extracts obtained, more or less highly concentrated solutions of the aromatic substances in ethanol or in mixtures of ethanol and water are obtained. This method for producing aromatic substances is first of all very time-consuming (taking 10—14 days), as a result of which correspondingly high ethanol losses are caused by the relative high ethanol vapour pressure. Furthermore, some of the ethanol is left behind in the plants or drugs separated, so that overall ethanol losses of the order of magnitude of at least 10% have to be expected. A further drawback of the known processes resides in the limited yield of aromatic substances, generally between 50 and 80. With the conventional processes an increase in the yield, which would be necessary if only because of the usually valuable raw materials, only appears feasible at the cost of a disproportionately high increase in outlay and in this case in the time consumed, the increase in yield thereby achieved being very small.

Attempts have already been made to speed up the process and at the same time to increase the yield by raising the pressure and/or temperature in whatever system is in use. In addition to the consequently still higher outlay the improvements obtainable within safe ranges are unsatisfactory in relation to the cost incurred, and any further increase in the aforementioned parameters usually has uncontrollable incidental effects and in some cases results in serious deterioration of quality, so that such methods have not gained a footing in practice. Neither is the continuous re-pumping of the starting mixture a satisfactory means of increasing the output in relation to the cost. It has also been suggested that the reaction should be regulated by the choice of the most suitable solvent for the extract concerned, likewise in order to accelerate the process and increase the yield, (West German Specification 2327466). The main disadvantages of the last mentioned process resides in the fact that most of the solvents proposed have a toxic effect and therefore have to be completely removed from the extract produced. The most serious disadvantage of this proposal, however, is that the use of toxic solvents of this kind is doubtless without exception contrary to the legislation in force regarding foodstuffs.

The purpose of the invention is to develop a process of which the application can considerably speed up the production of extracts especially suitable for the spirituous liquor industry while maintaining at least the same quality and at the same time increasing the yield, and which, maintaining at least equally high quality in the final product and avoiding any undesirable incidental effects, not only is capable of providing the maximum increase in the yield but also of considerably speeding up the process as a whole.

According to the invention there is provided "Claim 1". The process is generally performed at ambient temperature. The said substance to be extracted may be a solid at ambient temperature. Preferably the ratio of the extraction liquid to the said mixture is from 4:1 to 10:1 by weight.

The ultrasonic radiation generally has a frequency of 20—50 kHz, preferably of 20—40 kHz, preferably an acoustic power of 10—100W and the influence of ultrasonic radiation is preferably maintained for a period of 10—100 minutes.

The mixture to be treated is generally chosen from fruit, nuts, plants, herbs and drugs, and the extraction liquid generally comprises an alcohol, water or a mixture thereof.

The acoustic radiation generally takes place in a suitable vessel, such as a sonic appliance with fixed or movable built-in vibrators, the extraction liquid at the same time serving as a transmission medium for the ultrasonic energy.

Optimum results both in the yield and in the quality of the end product in a preferred form of the process of the invention where the extraction liquid is an alcohol/water mixture can be obtained by suitably planned regulation of the ratio of alcohol to water and of the ratio of the extraction liquid to the said substance, in conjunction with the relevant acoustic radiation parameters. related to the particular substance to be extracted, which said variations, particularly in multi-stage extractions, make it possible to improve the preferred extraction of certain positive aromatic substances while a suitable preliminary extraction performed in advance enables substances which would detract from the taste of the product to be isolated from the actual extraction. By selecting a suitable initial temperature in the extraction system the slight temperature rise in the system as a whole caused by the ultrasonic treatment can also be utilized for further improving the result of the extraction. It has also been found that with a preferred form of the inventive process when the same initial material is extracted more than once and aromatic substances are to be removed such aromatic substances can be almost completely removed from the initial material present, i.e. the treatment embodying the invention ensures that the usually valuable raw materials will be utilized in an optimum manner, the quality of the final product being improved or at all events maintained. It was also found that with a number

of initial substances multiple extraction is clearly more satisfactory than a correspondingly prolonged single extraction. In particular, a greater quantity of extraction liquid in proportion to the particular quantity of substance to be extracted provided further advantages, and this ratio can also be varied from one stage to the next.

Examples of the Invention:

The invention will be described below in greater detail by reference to certain examples, and the comparative examples given relate to the particular extraction procedures to which preference is given at the time.

(1) Production of Lime Extract

(1.1) Comparative Example.

200 g of lime chips are first pretreated with hot water or steam.

The chips are then macerated with 520 ml of ethanol and 510 ml of water for 10—14 days.

(1.2) Example of the Invention:

(a) 200 g of lime chips (of the same initial substance) were ultrasonically treated in one litre of water in a suitable ultrasonic vessel at a freqency of 34 kHz and an acoustic power of 50 W for 10 minutes. After the discharge of the water the chips were ultrasonically irradiated for 60 minutes in a mixture of 520 ml of ethanol and 510 ml of water with the same ultrasonic parameters. The initial temperature of the system was 20°C and the final temperature 35°C.

After the first extract had been obtained the same material underwent a second extraction under the same conditions.

(b) 150 g of lime chips were subjected to the same preliminary treatment as in Ex. (1.2) (a) and then treated for 60 minutes in 800 ml of ethanol and 1.2 litres of water at a frequency of 40 kHz and with an acoustic frequency of 50W. During the ultrasonic radiation the temperature increased from 22°C to 36.8°C. After the first extract had been obtained the same material was likewise subjected to a second extraction under the same conditions.

The extracts obtained in the manner described were tasted and also tested spectrophotometrically and chromatographically. In the first ultrasonic extract in each case (regardless of the quantity of liquid) the content of aromatic aldehydes (such as vanillin) and total aromatic substances (absorption at 280 mm) was found to be higher than with the extract obtained in the comparative example. In each case the values obtained with the second ultrasonic extract were approximately the same as those obtained with the comparative example.

The sampling by taste fully confirmed this analysis.

The comparison between the extracts obtained in Ex. (1.2) (a) and Ex. (1.2) (b) showed that for given taste values an increase in the ratio of liquid to solid substance enables the yield to be increased still further.

(2) Production of Apricot Extract

(2.1) Comparative Example.

300 g of dried apricots were treated with 500 ml of spirit (alcohol), 520 ml of water and 200 ml of distillate and macerated for 14 days, being stirred several times in the process.

(2.2) Example of the Invention.

The same starting material as in the comparative example was treated utrasonically for 1 hour in a suitable ultrasonic vessel with a frequency of 34 kHz and an ultrasonic intensity of 50W. The initial temperature of the system was 20°C, the final temperature after the end of the treatment being 35°C.

In the second extract, produced with the same initial quantities and ultrasonic parameters, a temperature rise from 24° to 39°C. occurred.

The comparative tasting showed that the first extract had a far more distinct "full-fruit" aromatic taste than the extract from the comparative example, while the second extract was merely equal to that obtained by the comparative example.

(3) Production of Nut Extract.

(3.1) Comparative Example.

500 g of roast and roughground hazel nuts were treated with 1.5 litres of ethanol and macerated for 14 days, being stirred a number of times in the process.

(3.2) Example of the Invention.

(a) 500 g of roast and ground hazel nuts, with 1.5 litres of ethanol, were ultrasonically irradiated for 50 minutes in an ultrasonic vessel with a frequency of 34 kHz and an acoustic power of 50W. The temperature rose from 20° to 36.2°C. A further extract from the same material was produced under the same conditions after the extraction of the first.

(b) 500 g of roast and ground hazel nuts, with 1.2 litres of ethanol and 300 ml of water, were ultrasonically irradiated for 60 minutes under the same conditions (34 kHz, 50W). The temperature rose from 20° to 39°C. After the first sample had been taken a second sample was produced from the nuts, the same parameters being adopted as before.

The sampling of the specimens by taste showed that even the second extract of each example (3.2(a) and (b)) indicated gave more satisfactory values that the extract from the comparative example. In the overall evaluation it was the extracts from Ex.(3.2)(b) that gave the best results.

The process according to the invention was also applied to the production of extracts from bitter oranges, orange peel, almond peel and prunes, under special handling conditions. All the extracts produced were likewise found to be of satisfactory taste when sampled.

The process enables use to be made of taste correctives from the same points of view as the processes hitherto customary, again with the additional advantage of enabling considerable quantities of material to be saved.

The process to which the invention relates can also be successfully used for the extraction of mixtures from plants or parts thereof, herbs or drugs.

(4) Production of Vermouth Extract.

(4.1). Comparative Example

250 g of Torino herb mixture are macerated for 10 days with 1 litre of a mixture of alcohol and water (in proportions of 50%) and then distilled with steam.

(4.2) Example of the Invention

(a) 250 g of Torino herb mixture is treated for 60 minutes with 2 litres of a mixture of alcohol and water (50%) at a frequency of 34 kHz and with an acoustic power of about 50W, in an ultrasonic vessel. This led to a temperature increase from 25°C to 40°C. After the first extract had been taken the total amount was restored by the addition of 1 litre of a 50%strength mixture of spirit (alcohol) and water and the ultrasonic treatment repeated.

(b) After the first extract has been produced on analogous lines to Ex. (4.2) (a) the total amount was restored by the addition of 1 litre of water and the ultrasonic treatment was carried out for 75 minutes at 34 kHz and 50W. The temperature increased from 28°C to 50°C.

The sampling showed that the first ultrasonic extract in each case had a fully aromatic fruity taste which was richer than that of the comparative example. The second ultrasonic treatment carried out in the example of the invention, with a high proportion of water, provided the bitter element obtained in the comparative example by the steam treatment. The result of the tests agrees with that obtained with the lime extracts, in which the ultrasonic treatment with water mainly extracts the bitter substances (e.g. tannic substances) while the aromatic substances are mainly extractible with alcohol. The exemplified processes illustrative of the invention indicate that any desired nuance in the taste to be obtained by varying the ratio of initial mixture 1 to initial mixture 2.

CLAIMS

1. An extraction process for extracting a substance from a mixture containing the substance comprising subjecting the mixture in the presence of an extraction liquid for the said substance to the influence of ultrasonic radiation, the extraction liquid serving as a transmission medium for the ultrasonic energy.

2. An extraction process according to Claim 1, which is performed at ambient temperature.

3. An extraction process according to Claim 1 or Claim 2, wherein the said mixture is a solid at ambient temperature.

4. An extraction process according to any preceding claim, wherein the ratio of the extraction liquid to the said mixture is from 4:1 to 10:1 by weight.

5. An extraction process according to any preceding claim, wherein the ultrasonic radiation has a frequency of 20—50 kHz.

6. An extraction process according to Claim 5, wherein the upper frequency limit is 40 kHz.

7. An extraction process according to any preceding claim, wherein the acoustic power of the ultrasonic radiation is 10—100W.

8. An extraction process according to any preceding claim, wherein the mixture is subjected to the influence of ultrasonic radiation for a period of 10—100 minutes.

9. An extraction process according to any preceding claim, wherein the mixture to be treated is chosen from fruit, nuts, plants, herbs and drugs.

10. An extraction process according to any preceding claim, wherein the extraction liquid comprises an alcohol, water or a mixture thereof.

11. An extraction process according to Claim 1, substantially as herein described and exemplified.

12. An extracted substance which has been obtained by the process claimed in any preceding claim.

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